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1 **Mechanical pretreatment of municipal biowaste to produce an aqueous**
2 **slurry dedicated to anaerobic digestion**

3

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16

17 **Abstract**

18 This study investigated a wet mechanical pretreatment to improve methane production by
19 anaerobic digestion from biowaste material by separating a biodegradable aqueous slurry
20 fraction (ASF) from a more recalcitrant particulate fraction (PF). Four source-sorted
21 municipal biowaste were studied, namely Household (HBW), Supermarket (SBW),
22 Restaurant (RBW) and Green Biowaste (GBW). The treatment consisted in soaking the
23 waste in water and then press the slurry through a grid with 3-mm openings to separate
24 the 2 fractions. Methane production of ASF and PF obtained from the 4 biowastes were

25 measured using the BMP protocol and compared to the potential of the respective
26 untreated biowaste.

27 Results were very different for GBW as compared to the other 3 BW. With GBW, which
28 was the most lignocellulosic of the BW studied, only 17% of the initial methane potential
29 was recovered in the ASF. The extraction was much better on the other biowastes and
30 increased in the following order: HBW (58%) \approx RBW (57%) < SBW (67%). The ASF
31 from these biowastes exhibited low total solids contents and high BMPs (416, 408 and
32 423 NL_{CH₄}.g⁻¹_{vs} for HBW, RBW and SBW respectively).

33 The experimental results obtained in this study therefore showed that wet pressing
34 separation was an efficient pretreatment to improve and facilitate methane production by
35 anaerobic digestion of biowaste such as HBW, RBW and SBW.

36

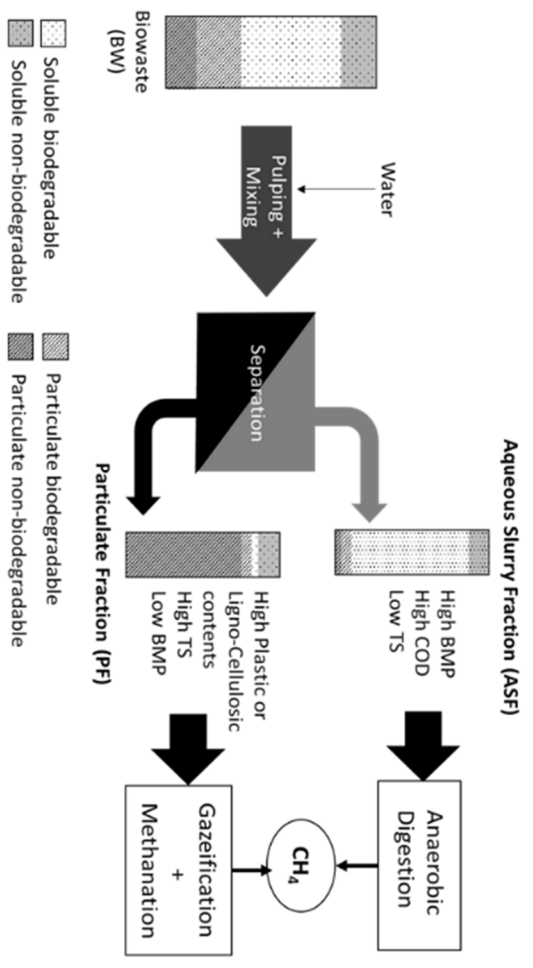
37 **Keywords:** biomethane potential; municipal organic waste; wet press separation; energy
38 recovery; biodegradable organic fraction; bioconversion

39

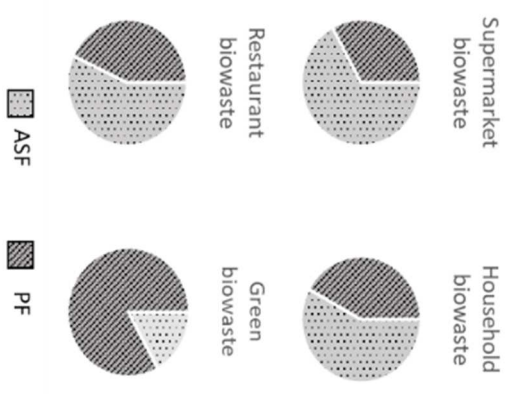
Graphical abstract

40 **Mechanical pretreatment of municipal biowaste to produce an aqueous**
41 **slurry dedicated to anaerobic digestion**

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43 Gourdon¹



Distribution of BMP



46 **1 Introduction**

47 Municipal solid waste production (MSW) is still growing in most European cities despite
48 the efforts made to reduce it (Fisgativa et al. 2016; Braguglia et al. 2018). MSW
49 comprises several categories of waste collected on urban territories, including biowaste
50 (BW). According to the European Directive 2018/851, BW includes biodegradable
51 garden and park waste, food waste from restaurants and supermarkets, and the
52 biodegradable organic fraction of household waste (European Union 2008). The
53 production of municipal biowaste in EU-28 was estimated to 86.10^6 Tons per year.
54 Household BW and Garden BW represented 60% and 35% of this production respectively
55 (European Environment Agency 2020). Due to their biological origins and specific
56 characteristics, BW are potential resources of organic matter for the production of
57 compost, nutrients, or energy (Escamilla-Alvarado et al. 2017).

58

59 Selective collection of BW is developing fast in several European urban areas (Bernstad
60 et al. 2014; Sidaine and Gass 2013). European regulations will make it compulsory in
61 2023 (European Union 2018). This evolution should improve the quality of the recovered
62 organic matter and thereby favor its reuse for agricultural purposes in the future (Hansen
63 et al. 2007). However, the possible presence of undesirable materials such as glass,
64 plastics, ceramics, etc and even hazardous substances such as heavy metals and other
65 micropollutants, is likely to remain a significant risk in big cities where the quality of the
66 selective collection may be degraded for various reasons (Weithmann et al. 2018). In this
67 context, energy recovery appears more adapted in urban territories because it would be
68 less sensitive to possible contaminations of the resource.

69

70 Among the different possible strategies to convert BW into energy, methane production
71 is attractive in urban territories. Methane has a high energy content of 10 kWh per Nm³,
72 several possible energy usages, and it is already distributed in many cities through
73 existing grids. BW however contains different proportions of biodegradable matter and
74 more recalcitrant constituents such as lignocellulosic compounds (St Joly et al. 2000;
75 Fisgativa et al. 2017). Several types of processes have therefore been used to optimize
76 methane production from BW. Biological anaerobic digestion is best adapted to convert
77 the humid and readily biodegradable organic constituents (Capson-Tojo et al. 2016).
78 Thermochemical processes such as gasification would be more adapted for their less
79 humid and more recalcitrant constituents (Vakalis et al. 2017). Pretreatments of BW are
80 therefore needed to separate the 2 types of constituents and apply adapted treatments to
81 each of them (Rodriguez-Valderrama et al. 2020).

82

83 Mechanical treatments have been used for decades in solid waste treatment applications,
84 and still are. Sorting operations are used to remove undesirable materials or, on the
85 contrary, recover valuable constituents from a waste flow (Ariunbaatar et al. 2014;
86 Pognani et al. 2012). Several authors have used mechanical operations as pretreatments
87 prior to anaerobic digestion (Hansen et al. 2007; Do Carmo Precci Lopes et al. 2019).
88 Press-separation was investigated to remove undesirable impurities and recalcitrant
89 particulate matter from BW (Jank et al. 2015) or to produce a biodegradable aqueous
90 slurry (Micolucci et al. 2015a; Do Carmo Precci Lopes et al. 2019). However, most of
91 published studies on the subject have concerned household BW mixed or not with green
92 waste. Do Carmo Precci Lopes et al. (2019) reported that the design and implementation
93 of mechanical pretreatments was still a challenging step to convert BW into methane

94 and/or compost. To our knowledge, no comparative study has been conducted on specific
95 BW categories as reported in the present article.

96

97 The present study investigated the implementation of wet pressing as a mechanical
98 pretreatment of 4 types of BW to optimize their anaerobic digestion. The treatment was
99 meant to generate an aqueous slurry fraction (ASF) expected to extract as much as
100 possible the soluble readily biodegradable constituents, and a particulate fraction (PF)
101 expected to collect fibrous and particulate constituents along with undesirable materials
102 that may be present. The aqueous slurry is to be oriented to anaerobic digestion to produce
103 methane, whereas the particulate fraction would be treated by gasification followed by
104 methanation of syngas.

105 **2 Materials and methods**

106 2.1 Selection and characterization of biowaste samples

107 The categories of BW material investigated in this study were selected in collaboration
108 with the technical services of Lyon Metropolitan Area, France, considering the following
109 criteria:

- 110 - Quantitative production on the considered territory;
- 111 - Availability (dispersion, accessibility), collection costs and possible difficulties;
- 112 - Territorial specificities, public policies, political strategies, other operational issues.

113 Four types of BW were selected, namely Garden BW (GBW) collected from domestic,
114 municipal and private sources; BW from restaurants (RBW) collected from private and
115 public collective catering sources including schools; Household kitchen BW (HBW)
116 collected from a set 200 hundred people (faculty members, staff and students of our

117 institution) who separated at source their own food waste; BW from supermarkets (SBW)
118 collected from a company specialized in depackaging of mixed supermarket food waste.
119 Representative samples of about 200 kg of each BW were collected in September 2018
120 following standard sampling guidelines and protocols NF EN 14899 (2006). Each sample
121 was homogenized by coarse shredding using a Blik BB350, and analyzed as reported in
122 a previous work (Moretti et al. 2020). All analyses were duplicated and results were well
123 reproducible. Table S1 (supplementary data) gathers the physico-chemical composition
124 and biodegradation potential of each BW, which were determined as described in Moretti
125 et al. (2020).
126

127 2.2 Biowaste pre-treatment by soaking and pressing

128 The protocol followed to treat the different BW samples is illustrated in Figure 1. Assays
129 were done in triplicates. Each shredded sample was soaked in de-ionized water in a 1 L
130 glass beaker. Two liquid to solid ratios were tested: 5 and 10 g of water per g of dry BW.
131 250 mL of the suspensions were mixed for 20 minutes at a room temperature of $23\pm 1^{\circ}\text{C}$,
132 in a Heidolph REAX 20 tumbler agitator set at 10 rpm. The objective of this operation
133 was to extract as much as possible the soluble and colloidal organic matter from the BW
134 samples.

135 Lab-scale filtration-compression cells were then used to separate ASF from PF. The
136 experimental setup is shown in Figure S1 (supplementary data). It was made of three
137 0.46L cylindrical stainless steel cells of 70 mm inner diameter and 120 mm height. A
138 stainless-steel disk perforated with holes of 3 mm diameter was placed at the bottom of
139 the cylinder. The BW suspensions obtained after soaking in water were introduced into
140 the 3 cells in order to run triplicate assays for each BW sample. The filtrate (ASF) was
141 collected from the bottom of the cells and its mass monitored online over time. After 20
142 minutes pressing, the system was stopped and PF was collected from the cells.

143

144 The experimental conditions tested are gathered in Table 1. Due to the high and regular
145 annual production of HBW and the current evolution of the European regulations which
146 will make it compulsory to collect this waste selectively and manage it specifically in
147 2023, HBW was selected as the resource to focus on in priority. The first two series of
148 experiments (first 2 lines of Table 1) were carried out only with the HBW. These
149 experiments compared 2 pressures (3 and 6 bars) and L/S ratios for the wet-pressing
150 treatment. The treatment duration was fixed at 20 min. Preliminary studies had shown
151 that this duration would guarantee no kinetic limitations that could bias the comparisons

152 between the waste samples. The last series of experiments was done to compare the results
153 obtained with the different waste samples. These experiments were carried out at a given
154 pressure (6 bars) and given L/S ratio (10). The four BW were studied for comparison.

155

156 2.3- Analysis of aqueous slurry (ASF) and particulate fractions (PF)

157

158 2.3.1 *Physical and chemical analyses*

159

160 ASF and PF obtained from the pretreatment of each BW were characterized in triplicates.

161 Total solids (TS) were measured by drying for 24h at 105°C known masses of samples
162 and weigh them dry (Baird and Bridgewater 2017). Volatile Solids (VS) were determined

163 as the mass loss upon calcination at 550 °C for 4 h (Baird and Bridgewater 2017).

164 Chemical oxygen demand (COD), Total Kjeldahl nitrogen (TKN), and ammonia nitrogen
165 (NH₃-N), were analyzed according to standard methods (Baird and Bridgewater 2017) on

166 samples dried at 70 °C for 3 days (until to constant weight) and crushed down to below 2

167 mm. Water soluble carbohydrates (WSC) and Volatile fatty acids (VFA) were analyzed

168 only in the aqueous slurries by Ionic Chromatography Dionex ICS5000 Thermo Fisher
169 after filtration at 0.45 µm.

170

171 2.3.2 *Biochemical methane potential (BMP)*

172

173 Biochemical methane potentials (BMP) were measured on each selected BW and on ASF
174 and PF, following the guidelines reported by Holliger et al. (2016). For BW samples and

175 PF, BMP assays were conducted at 35°C in 2 L glass vessels, for ASF, assays was

176 performed in 0.5 L. The inoculum was a fresh digested sludge taken from the wastewater

177 treatment plant of La Feyssine, Lyon, France (TS 2.0-3.3%wt; VS 1.4-2.2%wt). The
178 inoculum to substrate VS ratio was 2 g/g. Once filled, the glass vessels were purged with
179 N₂/CO₂ (80/20% v/v) gas for about 2 minutes, sealed and equilibrated at 35°C. Blanks
180 containing only the inoculum and water were systematically monitored along with each
181 series of assays in order to correct the recorded BMP from residual methane production
182 of the inoculum. Positive controls with cellulose were also monitored.

183 All blanks and assays were triplicated. Biogas production was followed by monitoring
184 the pressure in the flasks using a Digitron precision manometer. Biogas was released from
185 the flasks when the pressure exceeded 1200 hPa. Gas composition was analyzed using an
186 Agilent 3000 micro gas chromatography equipped with a thermal conductivity detector
187 (GC-TCD). Molsieve 5A (14 m length; pore size: 5 Å) and PoraPlot A (10 m length;
188 0.320 mm ID) columns were used as stationary phases for GC-TCD, using Argon as a
189 carrier gas. According to the protocol described by Holliger et al. (2016), tests were
190 stopped when the daily biogas production was less than 1% of the total volume of biogas
191 produced.

192 The rate of methane production was determined from the net methane production (i.e.,
193 after subtracting the blank methane production) according to a first order kinetic model
194 (equation 1).

$$V_{CH_4}(t) = V_{max}(1 - e^{-kt}) \quad (1)$$

195 where V_{CH_4} (NL) is the cumulated volume of methane produced at time t ; t (d) is the time,
196 V_{max} (NL) the maximum volume of methane produced, and the first order kinetic
197 constant.

198

199 2.3.4 Balance of total solids, volatile solids, oxidizable matter (COD) and BMP between
 200 aqueous slurry and particulate fraction

201

202 Mass balance calculations were performed on total solids (TS), volatile solids (VS), COD
 203 and BMP using the results obtained from the analyses done on each BW before treatment
 204 and on the fractions ASF and PF obtained after separation.

205 Parameters $x_{i,ASF}$ and $x_{i,PF}$ were defined as the proportions of parameter i (TS, VS, COD
 206 and methane production) of each respective BW sample which were transferred into ASF
 207 and PF fractions during the pretreatment operations. They were expressed in % w/w and
 208 calculated according to equations (2), (2'), (3), (3'), (4) and (4').

209

$$x_{VS,ASF} = \frac{m_{VS,ASF}}{m_{VS,ASF} + m_{VS,PF}} \cdot 100 \quad (2) \quad x_{VS,PF} = \frac{m_{VS,PF}}{m_{VS,ASF} + m_{VS,PF}} \cdot 100 \quad (2')$$

$$x_{CH_4,ASF} = \frac{V_{CH_4,ASF}}{V_{CH_4,ASF} + V_{CH_4,PF}} \cdot 100 \quad (3) \quad x_{CH_4,PF} = \frac{V_{CH_4,PF}}{V_{CH_4,ASF} + V_{CH_4,PF}} \cdot 100 \quad (3')$$

$$x_{COD,ASF} = \frac{m_{cod,ASF}}{m_{cod,ASF} + m_{cod,PF}} \cdot 100 \quad (4) \quad x_{COD,PF} = \frac{m_{cod,PF}}{m_{cod,ASF} + m_{cod,PF}} \cdot 100 \quad (4')$$

210 Where:

211 $m_{s,ASF}$: Mass of total or volatile solids in ASF (g_{TS} or g_{VS})

212 $m_{s,PF}$: Mass of total or volatile solids in PF (g_{TS} or g_{VS})

213 $V_{CH_4,ASF}$: Biomethane production measured on ASF (NL of CH₄)

214 $V_{CH_4,PF}$: Biomethane production measured on PF (NL of CH₄)

215 $m_{cod,ASF}$: Mass of COD measured in ASF (g_{O2})

216 $m_{cod,PF}$: Mass of COD measured in PF (g_{O2})

217

218 **3 Results and discussion**

219

220 3.1 Effects of pressure and L/S ratio on household biowaste (HBW) pretreatment

221 The effects of the operational conditions were investigated using this BW sample.

222 From preliminary experiments (not shown), three sets of conditions were compared: (i)

223 20 minutes soaking at a liquid to solid (L/S) ratio of 5 g of water per g of HBW dry solids

224 followed by pressing under 3 bars or (ii) 6 bars, (iii) 20 minutes soaking at a L/S ratio of

225 10 followed by pressing under 6 bars (Table 1).

226

227 Figure 2 shows the dynamics of extraction of ASF recorded during the pressing operation

228 of soaked HBW under the different operational conditions. Time 0 on the graphs showed

229 the time when pressure was applied. However, small volumes of solutions dripped out of

230 the cell by gravity before, and were included in the monitoring. The curves revealed 3

231 successive phases which resulted from the two processes involved in the operation. In a

232 first phase of 10 to 20 seconds corresponding to the filtration phase, the extracted mass

233 increased very sharply and linearly. According to the conventional theory of Ruth et al.

234 (1933a), which was verified by Vesilind (1994) on sludge material, filtration involves the

235 mobilization of free water under the effect of pressure and the convective transfer of the

236 suspended solids smaller than the size of the perforations in the filter.

237 A filtration cake consisting of the PF of HBW was thereby rapidly formed at the surface

238 of the filter disk by accumulation of the suspended solids larger than the disk openings

239 (see figure S2). This phenomenon rapidly reduced the permeability at the surface of the

240 disk and therefore the rate of ASF extraction slowed down over the next 1-2 minutes.

241 This was the second phase, corresponding to the formation and consolidation of the filter

242 cake. Particles in the filter cake were compressed, increasing furthermore the resistance
243 to mass flow. Finally, the last phase of the curves observed in the following 18-19 minutes
244 corresponded to the dewatering of the filtration cake. The extraction rate slowed down
245 progressively until no significant extraction was recorded, indicating that all free water
246 had then been removed from PF. At the completion of the assays (20 min), 69% of the
247 initial mass of soaked HBW was extracted at 6 bars and only 49 % at 3 bars for the ratio
248 of 5 g water.g⁻¹TS.

249 Increasing the pressure from 3 to 6 bars at a L/S ratio of 5 g_{water}.g⁻¹ TS increased the mass
250 extracted in the filtration phase from about 20% to about 35%. of the initial mass (Fig.
251 2). However, the curves over the last phase (dewatering of filter cake) were very similar
252 at 3 and 6 bars. Increasing the pressure also increased logically the TS content of PF from
253 26 to 41 % of the mass of the fraction (Table 2) since more aqueous solution was
254 extracted. TS content of ASF also increased slightly from 7.1 to 9.3 % probably due to
255 the extraction of dissolved and colloidal compounds.

256 Increasing the L/S ratio from 5 to 10 g_{water}.g⁻¹TS in the soaking operation of HBW
257 increased furthermore the extraction during the filtration phase under 6 bars from 69% at
258 L/S of 5 to 88% of the initial mass at L/S of 10. This result was explained by the higher
259 proportion of water in the initial soaked HBW, which was easily extracted in the filtration
260 phase. TS content in ASF was logically smaller at L/S of 10 as compared to L/S of 5
261 (Table 2). It can be noted that TS contents obtained here in the aqueous fraction (from 5
262 to 9.3 %, Table 2) were lower than reported in similar studies done on urban biowaste by
263 other authors where TS contents as high as 18 to 31% were reported (Jank et al. 2015; do
264 Carmo Precci Lopes et al. 2019; Micolucci et al. 2015b). This parameter however is
265 strongly modified by the experimental conditions, which vary from one study to another.

266 For example, do Carmo Precci Lopes et al. (2019) studied dry press separation with 12
267 mm openings, whereas Jank et al. (2015) used filters with 8 mm openings.

268

269 The mass proportions of each fraction produced from HBW were calculated by equations
270 1 and 1' (TS) and 2 and 2' (VS). Results are shown in Fig. 3.

271 Fig. 3a showed that the major part of the initial soaked waste mass was collected in ASF.

272 This was logical since this fraction received all the free water present in the initial soaked

273 waste. Increasing the pressure during the pressing operation allowed to extract more free

274 water and thereby increased the mass proportion of ASF. Increasing the liquid to solid

275 ratio in the soaking phase had the same effect for the same reason. Using a similar

276 approach as in this study, Jank et al. (2015) reported almost 85% ww extraction in the

277 aqueous fraction with a filtration disk perforated with 8 mm holes. Similar results were

278 reported by Novarino and Zanetti (2012) with extruded HBW at 8 mm holes. Hansen et

279 al. (2007) reported lower values with 55% to 64% ww extraction using an not-optimized

280 industrial screw press process.

281 Figs. 3b and c however showed that the dry matter (total solids, TS) initially present in

282 the soaked BW was collected mostly in PF. The organic matter (VS) followed the same

283 pattern as the total solids. Increasing the pressure from 3 to 6 bars in the pressing operation

284 or the L/S ratio in the soaking phase was found to increase the proportion of the VS

285 extracted into PF. This result was probably due to a stronger extraction of particulate

286 organic matter from the soaked waste into ASF by the increased amounts of free water

287 extracted into ASF under these conditions.

288

289 3.2 Pretreatment of the four biowaste by soaking at $10 \text{ g}_w \cdot \text{g}_{\text{TS}}^{-1}$ followed by pressing at 6

290 bars)

291

292 *3.2.1 Total mass, total solids and volatile solids distribution between aqueous slurry and*
293 *particulate fraction*

294

295 Figure 4 shows the dynamics of the extraction of ASF recorded in the operation of
296 pressing of the four soaked BW investigated.

297

298 The curves obtained with all waste samples exhibited a very sharp slope at the very
299 beginning of the treatment, corresponding to the first phase, or filtration phase, discussed
300 above, and a flat slope in the last phase corresponding to the filter cake dewatering
301 process. The second phase showed however two distinct patterns. With GBW and SBW
302 samples, phase 2 was almost not observed, whereas it occurred over 1 to 2 minutes with
303 RBW and HBW samples. This result was attributed at different characteristics in the
304 formation and consolidation of the filter cake. GBW probably generated a more porous
305 and less compressible filter cake than RBW, SBW and HBW, due to its higher contents in
306 lignocellulosic organic compounds. It was therefore probably more rapidly stable,
307 resulting in a very short phase 2. At the end of the treatment, 76% (GBW) to 90% (SBW)
308 of the initial mass of soaked waste was collected in ASF.

309

310 Figure 5 a, b and c shows the distribution of the overall mass, dry mass (TS) and organic
311 dry mass (VS) of the respective initial soaked waste between the two fractions produced.

312 TS concentrations in the fractions are given in Table 3.

313 Figure 5 revealed relatively similar results for SBW, HBW and RBW samples. For these
314 samples, 86 to 92 % of the initial wet mass of soaked waste was collected in ASF, and 39
315 to 52 % of TS or VS were collected in PF. GBW however exhibited a distinct behavior,

316 with more than 90 % of TS or VS collected in PF. This observation suggested that the
317 biochemical composition of the waste played a strong role. In a previous article, Moretti
318 et al. (2020) reported that GBW sample was composed of lignocellulosic materials at up
319 to 82% of VS. These biopolymers are very stable, recalcitrant, and poorly soluble. They
320 were therefore collected almost quantitatively in the PF. In contrast, the other BW studied
321 here mostly contained food products, with a higher proportion of less recalcitrant, more
322 soluble compounds. Part of the TS initially present in the BW were therefore degraded
323 and leached during soaking, and therefore smaller proportions were collected in PF.
324

325 *3.2.2 Distribution of methane potential in the 2 fractions obtained from each biowaste*

326

327 Table 4 shows the results from the BMP determinations of the fractions produced from
328 the pretreatment of each BW. Figure 6 illustrates the time course of methane production
329 recorded during the BMP assays.

330

331 Biogas production from either fraction obtained from HBW, RBW and SBW pretreatment
332 followed first order kinetics. No inhibition was observed. Methane productions from ASF
333 of these 3 BW were found in the same order of magnitude, ranging from 408 to 423
334 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$. These results were relatively close to previously published data. Micolucci
335 et al. (2015b) obtained a production of 470 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$ from the anaerobic digestion of
336 pressed HBW using a mesophilic digester. Jank et al. (2015) reported slightly smaller
337 methane potentials of 360 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$ on pressed biowaste using the BMP protocol. The
338 differences observed may be attributed to the different origins of the waste materials and
339 the different experimental protocols used.

340 The kinetic constants determined from the curves of biogas production were also very
341 close for the 3 biowaste HBW, RBW and SBW, ranging between 0.35 and 0.40 d^{-1} . These
342 observations suggested that the aqueous slurry fractions from these BW contained readily
343 biodegradable compounds in the forms of dissolved molecules, colloids and small
344 particles.

345 RBW and SBW samples exhibited similar methane productions from their respective PF
346 and ASF (between 408 and 455 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$), indicating that the soaking operation was
347 not efficient enough in extracting the readily biodegradable constituents from these BW
348 into the ASF. The corresponding kinetic constants however were smaller in PF than in

349 ASF (0.26 and 0.28 d⁻¹ vs. 0.35 and 0.40 d⁻¹), indicating that the PF fractions contained
350 more recalcitrant compounds than the corresponding ASF.

351 The efficiency of soaking operation was better with HBW, as shown by the lower BMP
352 of PF (269 ± 21 NL_{CH₄}.g⁻¹_{VS}) as compared to ASF (416 ± 13 NL_{CH₄}.g⁻¹_{VS}). This result
353 was attributed to the lignocellulosic contents, which was higher in HBW than in RBW
354 and SBW samples as shown in Table S1, making PF of HBW less readily biodegradable
355 than those of RBW and SBW.

356 Finally, GBW logically exhibited the smallest methane productions from each of its
357 fractions ASF and PF (160 and 60 NL_{CH₄}.g⁻¹_{VS}, respectively) as compared to all the other
358 waste tested. The kinetic constant recorded on its PF was the smallest of all, confirming
359 the recalcitrance of its ligno-cellulosic constituents. These observations were explained
360 by the nature, origin and composition of this BW (see Table S1).

361 Figure 5d shows the proportion of the volume of methane produced by ASF and PF from
362 each BW in the BMP assays, calculated by equations 3 and 3' (see materials & methods
363 section). It also shows the distribution of COD between each fraction of each waste,
364 calculated by eq. 4 and 4'.

365

366 The distributions of COD and methane production between the two fractions followed
367 very similar patterns for each BW. The efficiency of the pretreatment operations in
368 extracting as much as possible the methane potential of the BW materials into ASF was
369 found to decrease in the following order: SBW (67%) > HBW (58%) ≈ RBW (57%) >>
370 GBW (17%). This result was fairly well correlated to the distribution of volatile solids
371 (VS, see Fig. 5c), and very correlated to the distribution of COD (Fig. 5d). Working on a

372 mixture of HBW and GBW, do Carmo Precci Lopes et al. (2019) reported a higher
373 extraction of 82 % of methane potential in aqueous slurry.

374 In our study, the pretreatment operations allowed for SBW, HBW and RBW samples to
375 concentrate in ASF between half (48%) and more than the 2/3rds (71%) of COD and
376 methane productivity. In contrast, with GBW sample, the same operations conducted
377 under the same conditions concentrated strongly these parameters in the PF (83-94%).

378

379 *3.2.3 Composition of aqueous slurry fractions (ASF)*

380

381 Table 5 gathers the results from the chemical analyses of ASF obtained from the
382 pretreatment of each BW. It can be seen that the aqueous slurry fractions produced from
383 HBW, RBW and SBW exhibited very similar chemical compositions, except for N-TKN
384 concentration which was much smaller in ASF from HBW (177 mg/L) than those from
385 RBW and SBW (around 1000 mg/L). This may be attributed to a higher proportion of
386 meat products in RBW and SBW samples. The low pH (4.6 to 4.9) and relatively high
387 VFA concentrations (from 3.3. to 5.1 g_{COD}.L⁻¹, mainly as lactic and acetic acids),
388 suggested that some reactions of acidogenesis had already started within the biowaste
389 during the collection time and the pretreatment of these BW, thereby confirming their
390 high biodegradability.

391 The chemical composition of the ASF produced from GBW pretreatment was very
392 different from that of the ASF obtained from the other waste. It confirmed that GBW was
393 not adapted to anaerobic digestion.

394

395

396

397 3.2.4 Energy balance estimation

398

399 The results discussed above showed the possible added-value of implementing a wet-
400 press pretreatment prior to anaerobic digestion of biowaste with low lignocellulosic
401 content ($< 40\%$ of VS).

402 Further investigations at a larger scale and complementary economic considerations
403 appear however necessary to define the optimal conditions of implementation. Although

404 the nature and scale of the present study are actually not adapted to such estimations,
405 efforts were made to propose a simplified evaluation of the energy balance of the system.

406 Calculations are detailed in Table 6. The potential energy productions from anaerobic
407 digestion of the aqueous slurry fractions were calculated for a unit mas of organic matter

408 ($MJ.kg_{VS}^{-1}$) by multiplying the BMP values ($N_{LCH_4}.kg^{-1}_{VS}$) by the heat of combustion of
409 methane (35.9 kJ / L). The energy needed for the wet-press pretreatment was evaluated

410 in a simplified manner as the work developed to press the soaked waste under a constant
411 pressure of 6 bars ($6 \cdot 10^5$ Pa) over a volume variation ΔV equal to the difference between

412 the initial and the final volume. In addition, an overall energy yield of 0.3 was considered
413 between the electrical energy consumed and the work $-P \Delta V$ developed by the press. The

414 energy consumed for soaking the waste was neglected.

415 Table 6 shows that the energy consumed to run the wet-press pretreatment was very small
416 as compared to the energy potentially produced by anaerobic digestion ($>1\%$), with

417 exception for GBW, where the recovered amount of VS in the slurry was very small and
418 poorly degradable. In a first approach, it can therefore be considered that the

419 implementation of the pre-treatment is energetically acceptable.

420

421

422 **4 Conclusions**

423 A wet mechanical pretreatment was designed to improve and facilitate methane
424 production from biowaste. The treatment consisted in separating a biodegradable aqueous
425 slurry fraction from a more recalcitrant particulate one. Pressing of soaked waste was
426 investigated and shown as a good approach according to the experimental results
427 obtained.

428 The extraction of the biodegradable organic matter into the aqueous slurry fractions ASF
429 was found better with the biowaste materials containing relatively little amounts of
430 lignocellulosic substances. The efficiency of the pretreatment operations in extracting
431 methane potential of the BW materials into ASF was found to decrease in the following
432 order: SBW (67%) > HBW (58%) \approx RBW (57%) \gg GBW (17%). The pretreatment
433 operations were therefore poorly efficient with respect to this criterion for waste material
434 with high lignocellulosic contents such as Green BW. Energy balance of the system was
435 highly positive considering energy production from biogas and energy demand for press-
436 filtration. Further investigations are in progress to scale up the results.

437 **Supplementary data**

438 E-supplementary data for this work can be found in e-version of this paper online

439

440 **Declarations**

441 Ethics approval and consent to participate

442 Not applicable

443 Consent for publication

444 Not applicable

445 Availability of data and materials

446 The datasets used and/or analysed during the current study are available from the
447 corresponding author on reasonable request.

448 Competing interests

449 The authors declare that they have no competing interests

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453

454 Authors' contributions

455 PM and MMO have conducted the experimental work, the analyses, most of data
456 interpretation and draft redaction of the article.

457 RB was the project coordinator, the major contributor to the design of experimental
458 work, and contributor to the redaction of the manuscript

459 PB and RG were major contributors in the research project and in the redaction of the
460 manuscript.

461 JMA and ABC contributed in redaction of the manuscript

462 All authors have read and approved the final manuscript.

463

464

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475 **References**

- 476 Ariunbaatar J, Panico A, Esposito G, Pirozzi F, Lens PNL (2014) Pretreatment methods
477 to enhance anaerobic digestion of organic solid waste. *Applied Energy* 123:143-156.
- 478 Baird R, Bridgewater L (2017) Standard methods for the examination of water and
479 wastewater. 23rd edition. Washington D.C. American Public Health Assoc.
- 480 Bernstad A (2014) Household food waste separation behavior and the importance of
481 convenience. *Waste Manage* 34:1317-1323.
- 482 Braguglia C, Gallipoli A, Gianico A, Pagliaccia P (2018) Anaerobic bioconversion of
483 food waste into energy : A critical review. *Bioresour Technol* 248:37-56
- 484 Capson-Tojo G, Rouez M, Crest M, Steyer JM, Delgenes JP, Escudié R (2016) Food
485 waste valorization via anaerobic processes: a review. *Environ Sci Biotechnol* 15:499-
486 547.
- 487 Do Carmo Precci Lopes A, Robra S, Müller W, Meirer M, Thumser F, Alessi A, Bockreis
488 A (2019) Comparison of two mechanical pre-treatment systems for impurities
489 reduction of source-separated BW. *Waste Manage* 100:66-74.
- 490 Escamilla-Alvarado C, Poggi-Varaldo HM, Ponce-Noyola MT (2017) Bioenergy and
491 bioproducts from municipal organic waste as alternative to landfilling: a comparative
492 life cycle assessment with prospective application to Mexico. *Environ Sci Pollut Res*
493 24:25602–25617.
- 494 European Environment Agency (2020) Bio-waste in Europe – turning challenges into
495 opportunities. EEA Report 04/2020. ISSN 1977-8449.
- 496 European Union (2008) Directive 2008/98/EC of the European Parliament and the
497 Council of 19 November 2008 on Waste and Repealing Certain Directives. Official J
498 of the European Union 22/11/2008.

499 European Union (2018) Directive 2018/851 of the European Parliament and the Council
500 of 30 May 2018, amending Directive 2008/98/EC on waste. Official J of the European
501 Union 14/06/2018.

502 Fisgativa H, Tremier A, Dabert P (2016) Characterizing the variability of food waste
503 quality: A need for efficient valorisation through anaerobic digestion. *Waste Manage*
504 50:264-274.

505 Fisgativa H, Tremier A, Le Roux S, Bureau C, Dabert P (2017) Understanding the
506 anaerobic biodegradability of food waste: Relationship between the typological,
507 biochemical and microbial characteristics. *J Environ Manage* 188:95-107.

508 Hansen TL, Jansen JLC, Davidsson A, Christensen TH (2007) Effects of pre-treatment
509 technologies on quantity and quality of source-sorted municipal organic waste for
510 biogas recovery. *Waste Manage* 27:398-405.

511 Holliger C, Alves M, Andrade D, Angelidaki I, Astals S, Baier U, Bougrier C, Buffière
512 P, Carballa M, Wilde V, Ebertseder F, Fernández B, Ficara E, Fotidis I, Frigon JC ,
513 de Laclos HF, Ghasimi DSM, Hack G, Hartel M, Heerenklage J, Horvath IS, Jenicek
514 P, Koch K, Krautwald J, Lizasoain J, Liu J, Mosberger L, Nistor M, Oechsner H,
515 Oliveira JV, Paterson M, Pauss A, Pommier S, Porqueddu I, Raposo F, Ribeiro T,
516 Pfund FR, Strömberg S, Torrijos M, Eekert MV, Lier JV, Wedwitschka H, Wierinck
517 I (2016) Towards a standardization of biomethane potential tests. *Water Science*
518 *Technol* 74:2515-2522.

519 Jank A, Müller W, Schneider I, Gerke F, Bockreis A (2015) Waste Separation Press
520 (WSP): A mechanical pretreatment option for organic waste from source separation.
521 *Waste Manage* 39:71-77.

522 Micolucci F, Gottardo M, Malamis D, Bolzonella D, Pavan P, Cecchi F (2015a) Analysis
523 of Meso/Thermo AD process applied to pressed biowaste. *Waste biomass valor*
524 *6:723–731*.

525 Micolucci F, Gottardo M, Cavinato C, Pavan P, Bolzonella D (2015b) Mesophilic and
526 thermophilic anaerobic digestion of the liquid fraction of pressed BW for high energy
527 yields recovery. *Waste Manage* 48:227-235.

528 Moretti P, Morais de Araujo J, Borges de Castilhos A, Buffiere P, Gourdon R, Bayard R
529 (2020) Characterization of municipal biowaste categories for their capacity to be
530 converted into a feedstock aqueous slurry to produce methane by anaerobic digestion.
531 *Sci Total Environ* 726:137084.

532 NF EN 14899 (2006) Characterization of waste - Sampling of waste materials -
533 Framework for the preparation and application of a sampling plan.

534 Novarino D, Chiara Zanetti M (2012) Anaerobic digestion of extruded OFMSW.
535 *Bioresour Technol* 104:44-50.

536 Pognani M, Barrena R, Font X, Sanchez A (2012) A complete mass balance of a complex
537 combined anaerobic/aerobic municipal source-separated waste treatment plant. *Waste*
538 *Manage* 32:799-805.

539 Rodríguez-Valderrama S, Escamilla-Alvarado C, Rivas-García P, Magnin JP, Alcalà-
540 Rodríguez M, Garcia-Reyes RB (2020) Biorefinery concept comprising acid
541 hydrolysis, dark fermentation, and anaerobic digestion for co-processing of fruit and
542 vegetable wastes and corn stover. *Environ Sci Pollut Res* 27:28585–28596.

543 Ruth BF, Montillon GH, Motonna RE (1933a) Studies in filtration: I. Critical analysis of
544 filtration theory. *Ind Eng Chem* 25:76-82.

545 Sidaine JM, Gass M (2013) État de l’art de la collecte séparée et de la gestion de proximité
546 des biodéchets. *Awiplan ADEME*.

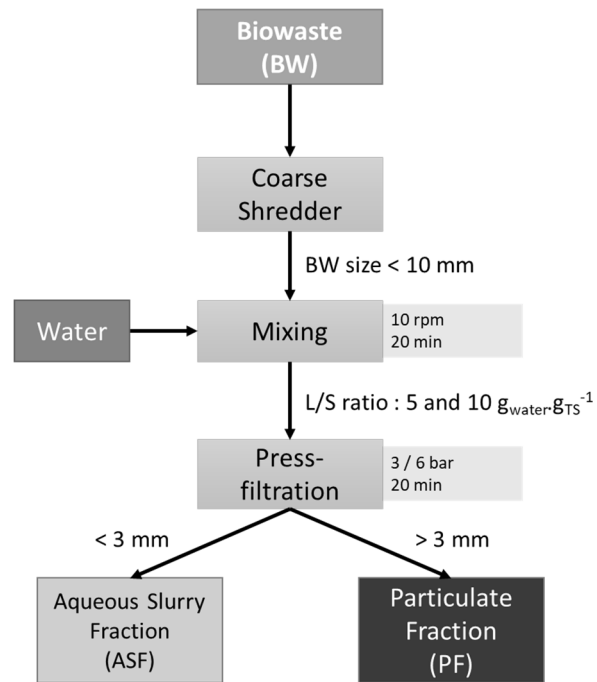
- 547 Vakalis S, Sotiropoulos K, Moustakas D, Malamis K, Vekkos M, Baratieri M (2017)
548 Thermochemical valorization and characterization of household BW. J of Environ
549 Manage 203:648-654.
- 550 Vesilind PA (1994) The role of water in sludge dewatering. Water Environ Res 66:4-11.
- 551 Weithmann N, Möller J, Löder M, Piehl S, Laforsch C, Freitag R (2018) Organic fertilizer
552 as a vehicle for the entry of microplastic into the environment. Sci Advance
553 4:eaap8060.

554 **Abbreviation List**

555	ASF	Aqueous Slurry Fraction
556	BMP	Biomethane Potential
557	BW	Biowaste
558	COD	Chemical Oxygen Demand
559	GBW	Green Biowaste
560	HBW	Household Biowaste
561	L/S	Liquid to Solid ratio
562	PF	Particulate Fraction
563	RBW	Restaurant Biowaste
564	SBW	Supermarket Biowaste
565	TS	Total Solid
566	VS	Volatile Solid
567		
568		

569 **Figures and Tables :**

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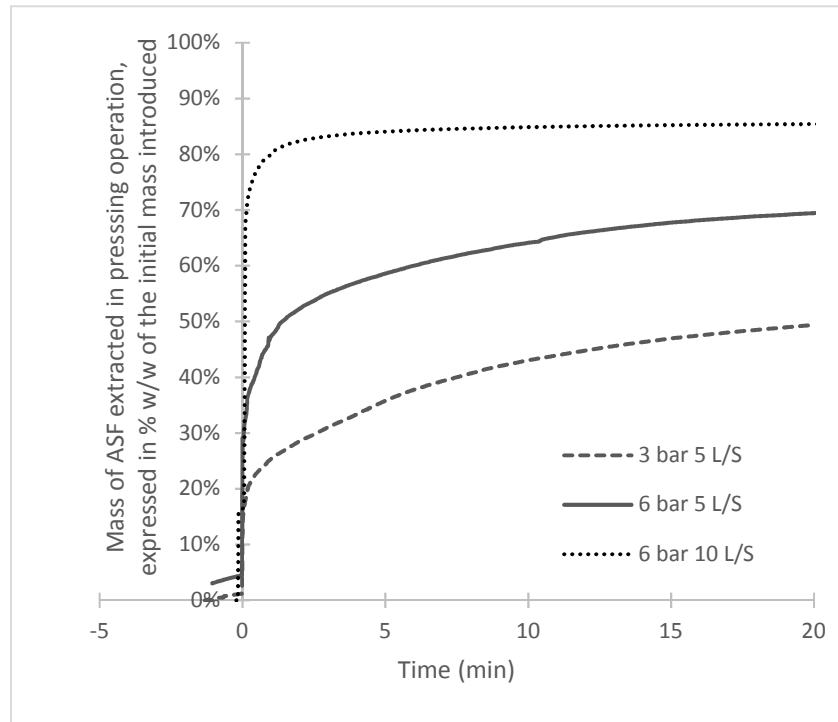


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573 **Figure 1** Flow chart of the pretreatment operations applied to each BW sample.

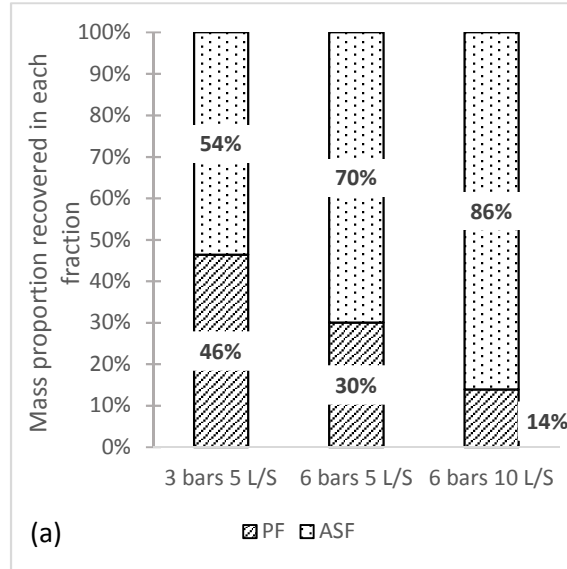
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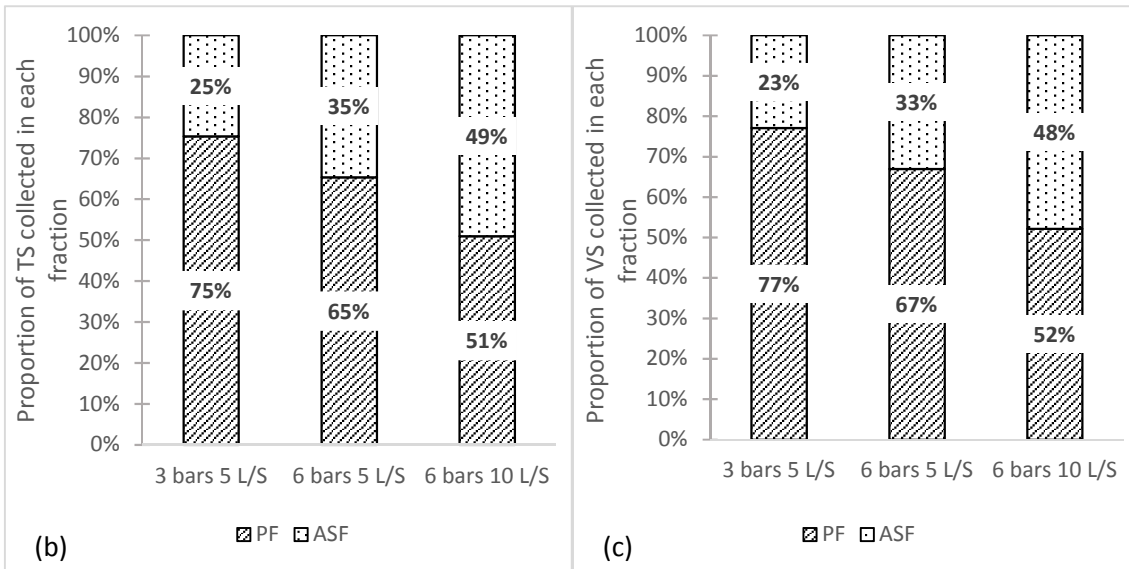
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576 **Figure 2** Time course of ASF extraction during the operation of pressing of soaked
 577 household BW (HBW) under different operational conditions: Soaking stage at L/S of 5
 578 $\text{g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$ followed by pressing at 3 bars or 6 bars; soaking at L/S 10 followed by pressing
 579 under 6 bars.

580



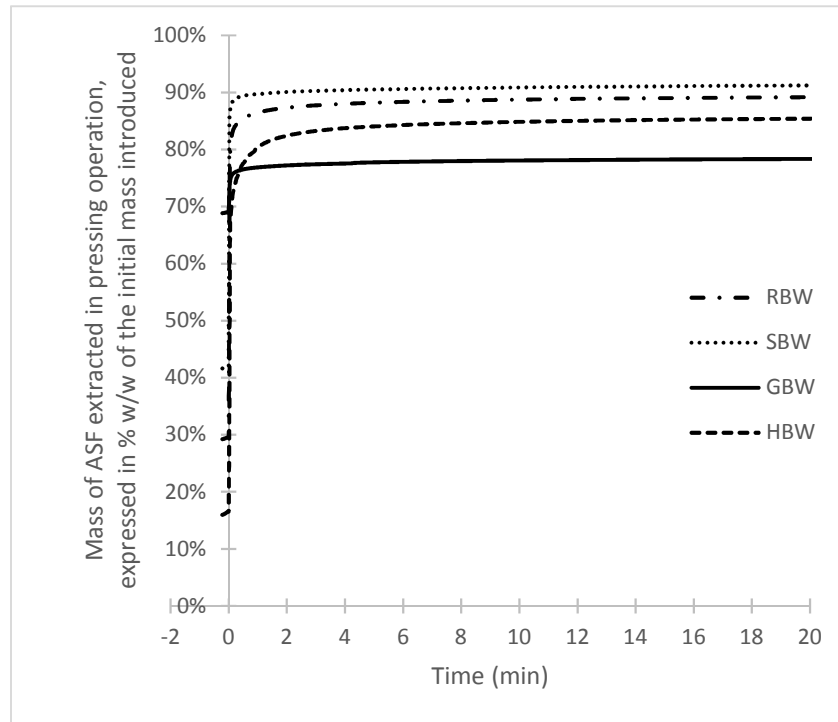
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583 **Figure 3** Total mass (a), total solids (b) and volatile solids (c) distributions between ASF
 584 and PF for HBW pretreatment under different experimental conditions. Calculations were
 585 done on the basis of 100 g ww / TS / VS of household BW (HBW) before pretreatment.

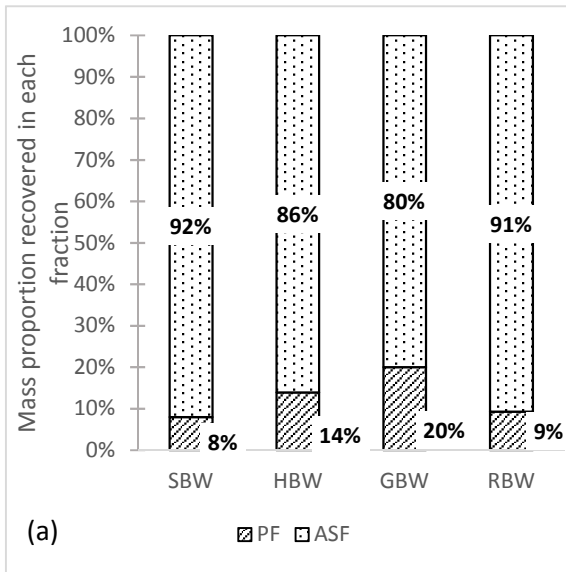
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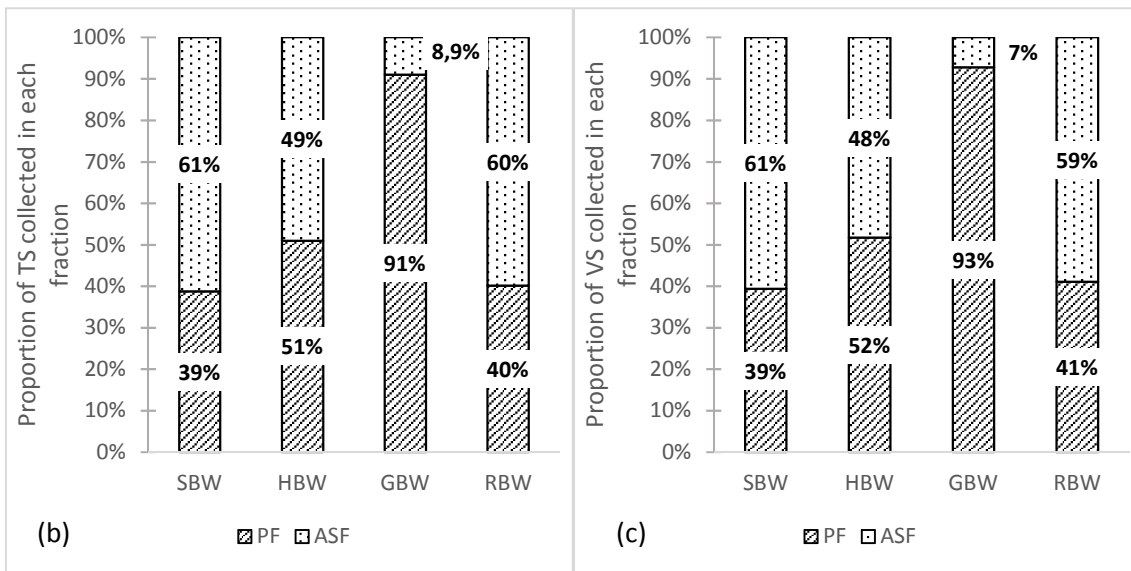
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588 **Figure 4** Time course of ASF extraction during the operation of pressing at 6 bars of the
 589 four BW previously soaked in water for 20 min at L/S ratio of $10 \text{ g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$. HBW:
 590 Household BW; RBW: Restaurant BW; SBW: Supermarket BW; GBW: Green BW.

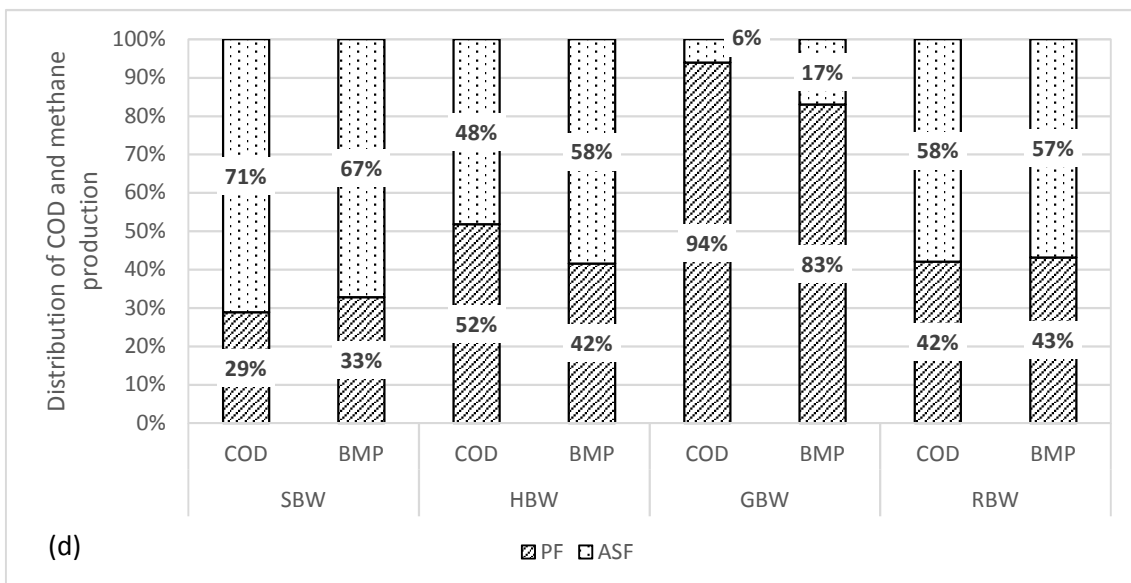
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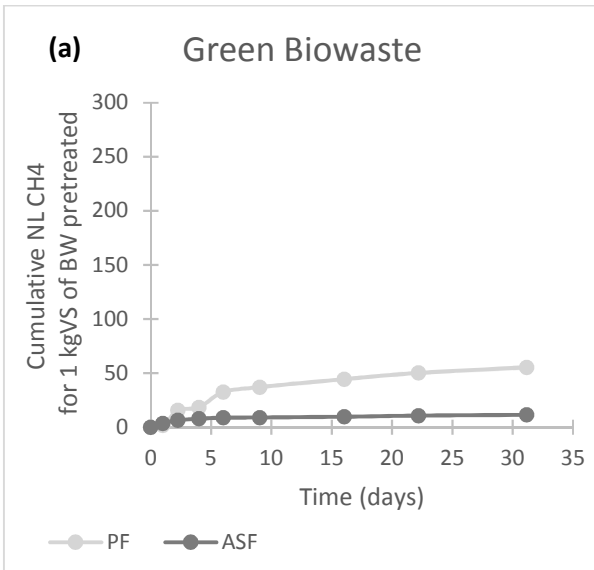


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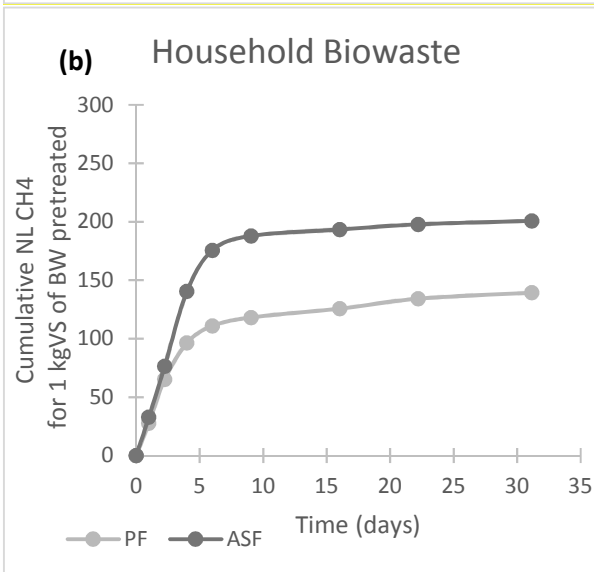
595 **Figure 5** Distribution of Total mass (a), total solids (b), volatile solids (c), COD and
596 methane production (d) between ASF and PF produced from the operation of pressing at
597 6 bars of the 4 BW previously soaked in water for 20 min at a L/S ratio of $10 \text{ g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$.
598 Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant
599 BW (RBW).
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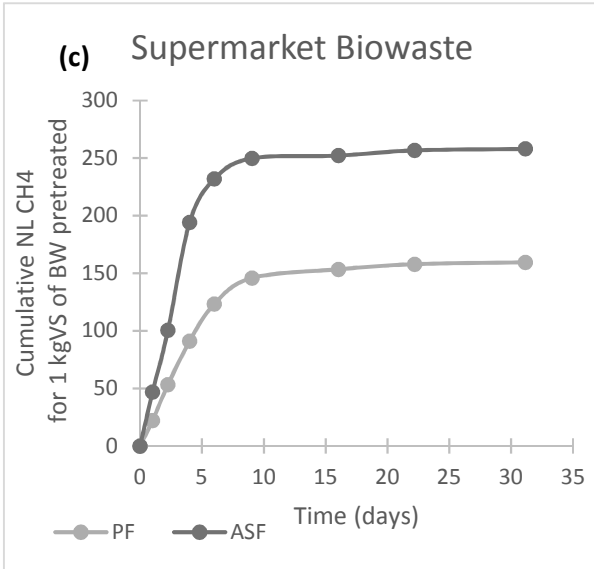
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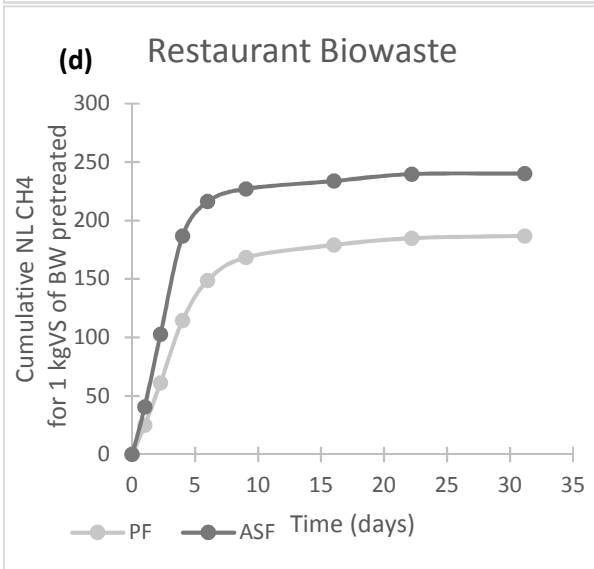
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608 **Figure 6** Production of methane of ASF and PF after pretreatment of 1 kgvs of GBW
 609 (a), HBW (b), SBW (c), RBW (d). Results are average values of BMP triplicate tests.

610

611

612

613 **Table 1** Experimental conditions used for the pretreatment operations of the BW samples.

Test	BW	L/S Ratio	Pressure	Pressing time
reference	tested	$(g_{water} \cdot g^{-1}_{TS})$	(bar)	(min)
3 bar 5 L/S	HBW	5	3	20
6 bar 5 L/S	HBW	5	6	20
	HBW			
6 bar 10 L/S	RBW	10	6	20
	SBW			
	GBW			

614 *HBW: Household BW, RBW: Restaurant BW, SMBW: Supermarket BW, GBW: Green BW*

615

616 **Table 2** TS contents of ASF and PF obtained from household BW (HBW) pretreatment
617 under different experimental conditions. Results are expressed in % of the dry mass of
618 the respective fraction. The results are average values and standard deviation of triplicated
619 analyses.

Experimental conditions			
<i>Fraction</i>	3 bar 5 L/S	6 bar 5 L/S	6 bar 10 L/S
Aqueous slurry (ASF)	7.1 ± 0.3	9.3 ± 0.6	5.0 ± 0.1
Particulate (PF)	26.0 ± 3.6	41.0 ± 3.5	32.2 ± 1.9

620

621

622 **Table 3** TS contents of ASF and PF after the operation of pressing at 6 bars of the four
 623 BW previously soaked in water for 20 min at a L/S ratio of 10 g_{water} .g⁻¹_{TS} . Results are
 624 expressed in % of the mass of the respective fraction.

Biowaste				
<i>Fraction</i>	HBW	RBW	SBW	GBW
Aqueous slurry (ASF)	5.0 ± 0.1	6.0 ± 0.1	6.0 ± 0.1	0.9 ± 0.1
Particulate (PF)	32.2 ± 1.9	39.2 ± 0.8	42.8 ± 2.0	40.0 ± 0.6

625 *Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant BW (RBW)*

626

627

628

629 **Table 4** BMP results (methane productions and kinetic constants) of ASF and PF
630 produced from the operation of pressing at 6 bars of the four BW previously soaked in
631 water for 20 min at a L/S ratio of 10 $\text{g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$. Results are average values of triplicated
632 analyses.

	HBW	RBW	SBW	GBW
Aqueous slurry (ASF)				
BMP ($\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{VS}$)	416 ± 13	408 ± 12	423 ± 13	160 ± 10
k (d^{-1})	0.35 ± 0.07	0.40 ± 0.01	0.35 ± 0.04	0.38 ± 0.02
Particulate (PF)				
BMP ($\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{VS}$)	269 ± 21	455 ± 18	409 ± 28	60 ± 6
k (d^{-1})	0.32 ± 0.01	0.28 ± 0.09	0.26 ± 0.03	0.14 ± 0.03

633

Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant BW (RBW)

634

**Calculated as the product of BMP value expressed in $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{VS}$ by the heating value of methane 35.9 kJ/NL*

635

636 **Table 5** Composition of the aqueous slurry fraction (ASF) produced from the operation
 637 of pressing at 6 bars of the four BW previously soaked in water for 20 min at a L/S ratio
 638 of 10 $\text{g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$. The results are average values of triplicated analyses.

Parameter	Unit	HBW	RBW	SBW	GBW
pH	-	4.9 ± 0.1	4.6 ± 0.1	4.9 ± 0.2	6.3 ± 0.1
N-NH ₃	$\text{mg}_N \cdot \text{L}^{-1}$	69 ± 0.2	73 ± 0.7	93 ± 1.3	16 ± 0.1
N-TKN	$\text{mg}_N \cdot \text{L}^{-1}$	177 ± 21	977 ± 27	1012 ± 13	69 ± 2
COD	$\text{g}_{\text{COD}} \cdot \text{kg}_{\text{TS}}^{-1}$	1067 ± 3	1056 ± 3	1056 ± 2	610 ± 1
VFA	$\text{equivalent g}_{\text{COD}} \cdot \text{L}^{-1}$	4.5 ± 0.6	3.3 ± 0.4	5.1 ± 0.8	0.6 ± 0.1
	<i>1</i>				
	% of COD	8.5	5.3	8.1	9.4
WSC	$\text{equivalent g}_{\text{COD}} \cdot \text{L}^{-1}$	10.2 ± 1.2	3.3 ± 0.1	7.5 ± 0.8	>0.1
	<i>1</i>				
	% of COD	19.3	5.3	12.0	0.3

639 COD: Chemical Oxygen Demand; VFA: Volatile Fatty Acids; WSC: Water Soluble Carbohydrates; TKN:
 640 Total Kjeldahl Nitrogen. Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant BW
 641 (RBW)

642
 643

644 **Table 6** Estimations of the energy balance of the wet pressing pretreatment in the process
 645 of anaerobic digestion of the 4 biowaste

	HBW	RBW	SBW	GBW
Potential energy production from aqueous slurry				
(ASF)				
BMP (NL _{CH4} .kg ⁻¹ vs)	416	408	423	160
Potential energy production* (kJ.kg ⁻¹ vs)	14930	14650	15150	5740
Estimated energy demand of wet pressing operation				
Wet pressing Pressure (in Pa)	6 10 ⁵	6 10 ⁵	6 10 ⁵	6 10 ⁵
Volume variation (in L)	0.43	0.45	0.45	0.41
Mass of sample in press (in g vs)	9.3	12.3	12.2	1.20
Energy yield of electrical press (dimensionless)	0.3	0.3	0.3	0.3
Estimated energy demand for pressing (kJ.kg ⁻¹ vs)	93.1	72.4	73.9	683
Estimated energy demand for pressing (% of energy produced)	0.6	0.5	0.5	12

646 *Calculated as the product of BMP value expressed in NL_{CH4}.g⁻¹vs by the heating value of methane 35.9 kJ/NL

647

648